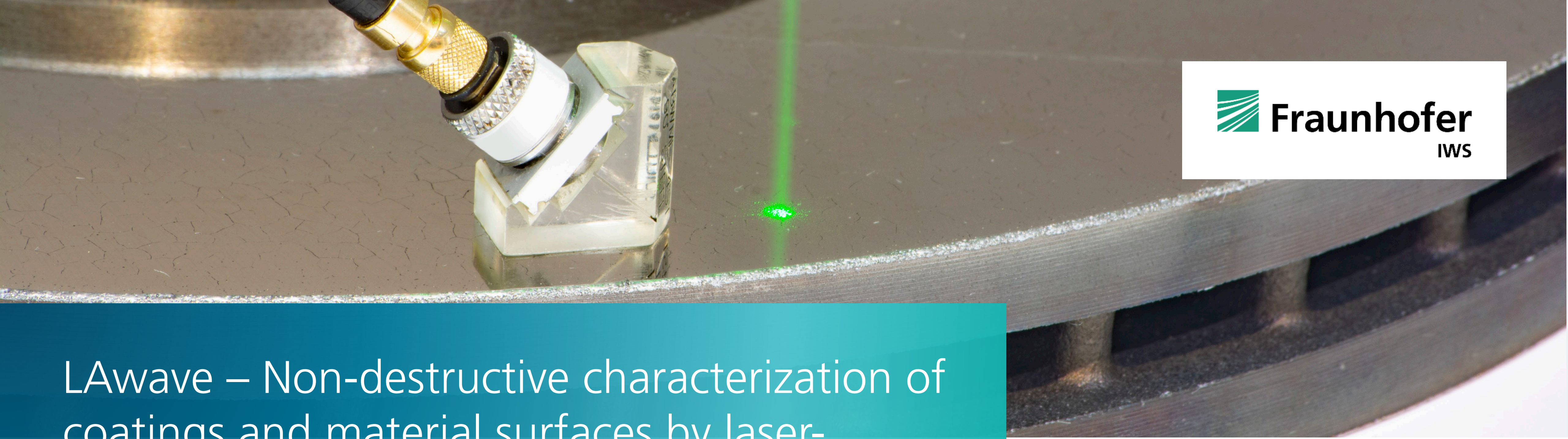
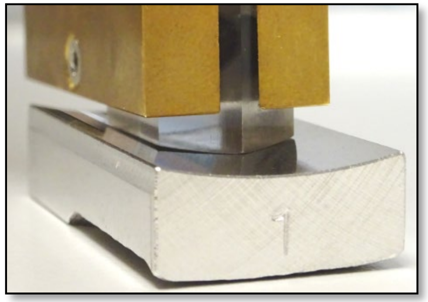
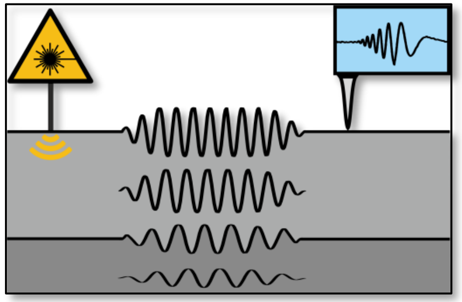
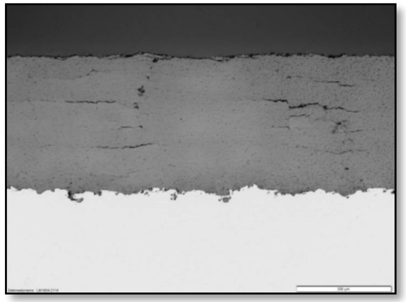


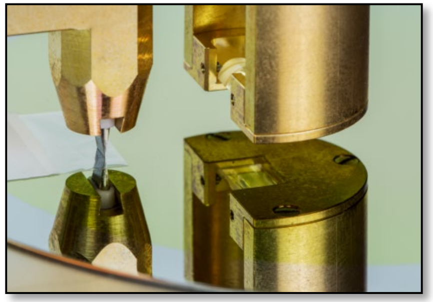
LAwave – Non-destructive characterization of coatings and material surfaces by laser-induced surface acoustic wave spectroscopy

Fraunhofer Institute for Material and Beam Technology IWS, Germany

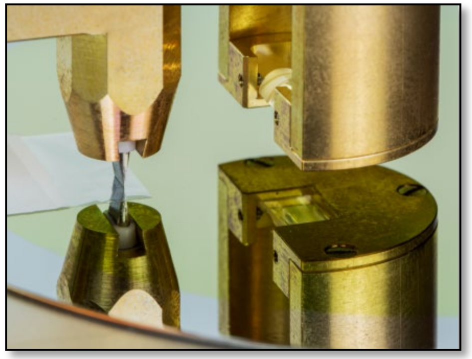


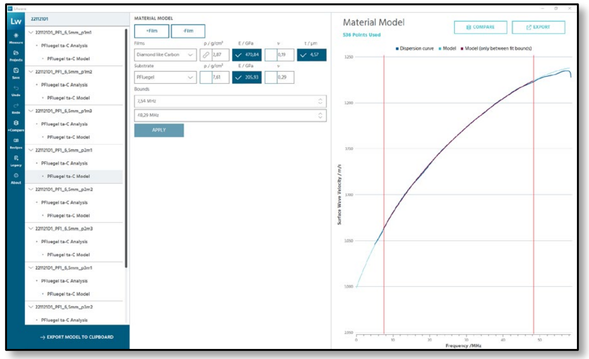






LAwave – at a glance





LAwave – Our one-of-a-kind measurement technology offers

- Access to surface material properties: Non-destructive, quick with highest accuracy
- For academia: unique research options for material science and solid state physics
- For industry: Easy quantification of surface properties in less than one minute
- Custom solutions for research, quality control, analysis and automation
- Fully integrated software for measurement and analysis

Facts and numbers

Complies with EN 15042-1:2006
30+ systems world wide
30+ years of experience
70+ peer reviewed contributions
2000+ citations
R&D 100 award

Contents

Introduction

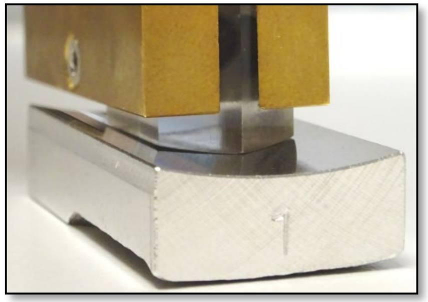
Components and technologies

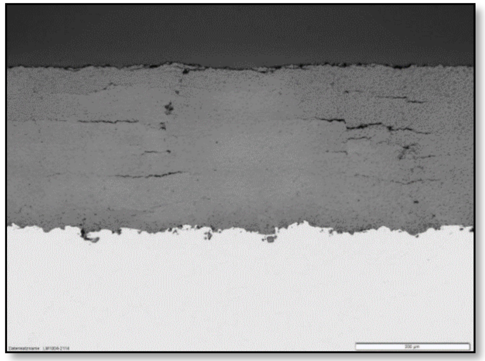
Mechanical properties of coated components

- Cylinder liner coatings (APS, wire arc spraying, …)
- Electric heaters (thermal sprayed coatings)
- Brake disk coatings (laser cladding)
- Heavy duty gear parts (cemented carbide coatings)
- 3D-printed metal components (SLM)
- Piston pins, tappets, chain components (PVD)
- And many more….

For R&D and quality control

- Effective Modulus (Pores, cracks, voids, delamination)
- Thickness
- Homogeneity
- Fast and effective – high throughput screening





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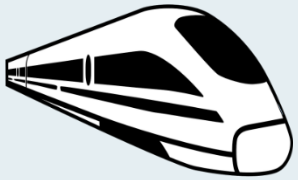
Highlights

John Caserta, CC BY 3.0

non-destructive



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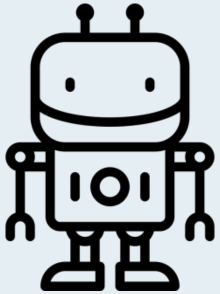
fast
(< 60 s)



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fully
automatable

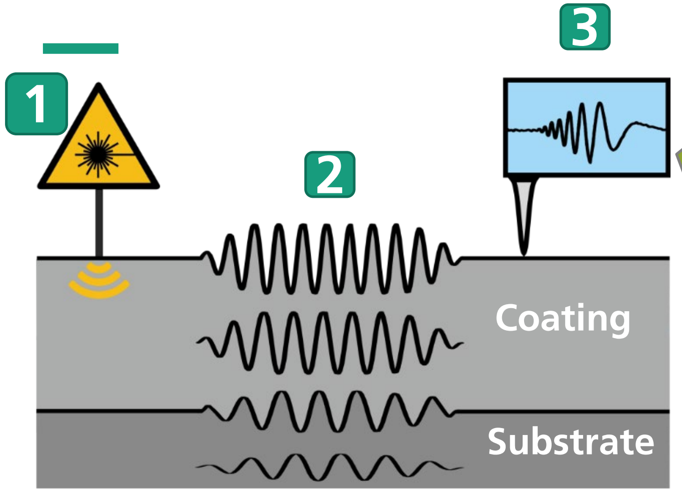
Basics

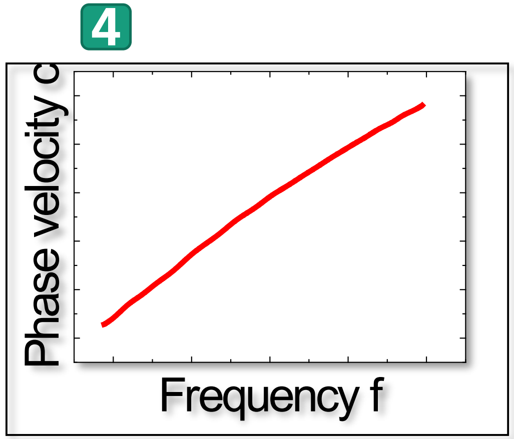
- LAwave® - Laser-induced surface acoustic waves spectroscopy
- Can access mechanical properties of coatings and surfaces
- Integral and effective mechanical information
- including pores, cracks and delamination
- Numerous applications for industrial quality control and R&D

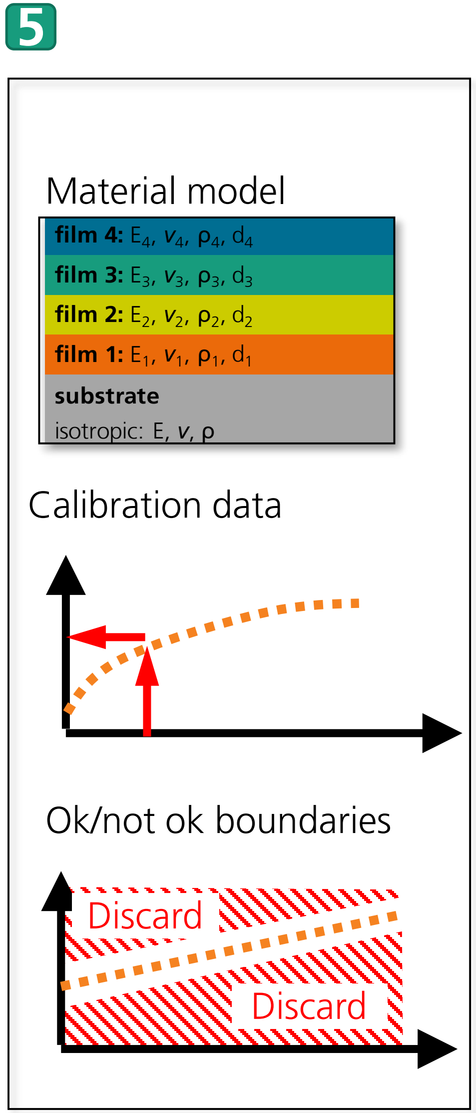
Advantages over indentation

- Faster measurement, no calibration, less consumables
- Higher precision, more and integral information
- Measures on rough surfaces
- True effective modulus: no plastic deformation, no compression of cracks, pores and defects

How it works - Overview







Possible results

- Young's Modulus
- Density
- Depth of: Nitriding layer, Case-hardening, Damage layers
- Porosity, crack density
- Delamination
- Build-up structure
- Surface hardness

…. Anything that affects your mechanical integrity of the material

(1) Broadband surface acoustic waves (SAW) induced by short laser pulses

(2) SAW propagation, velocity depends on frequency

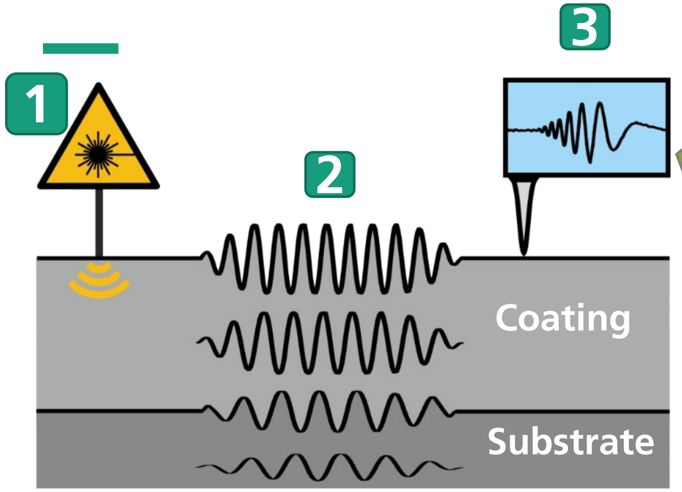
(3) SAW measurement: piezoelectric element → digitizing oscilloscope

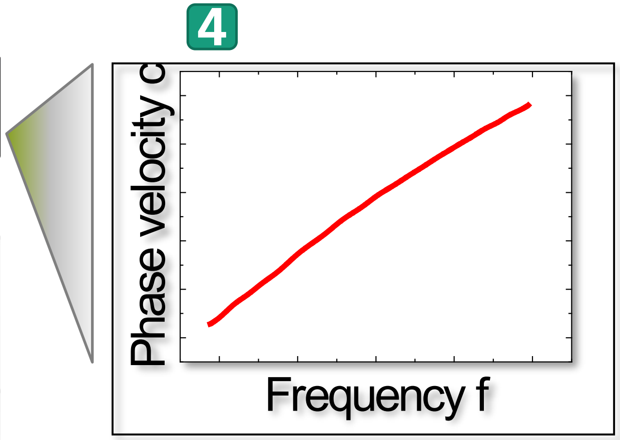
(4) Fourier transformation yields velocity over frequency (dispersion curve)

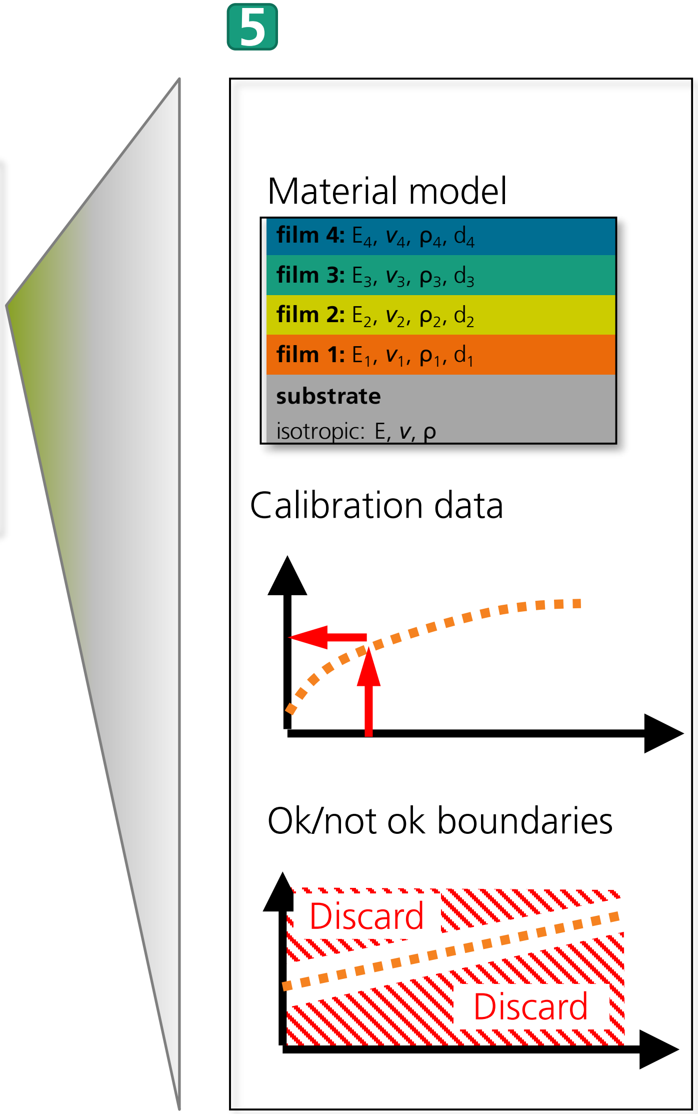
(5) Dispersion curve analysis using different evaluation strategies

Method

Method Overview







(1) Surface acoustic wave (SAW) generation

(2) SAW propagation through measured material volume

(3) SAW measurement by piezoelectric element

(4) Calculation phase velocity over frequency (dispersion curve)

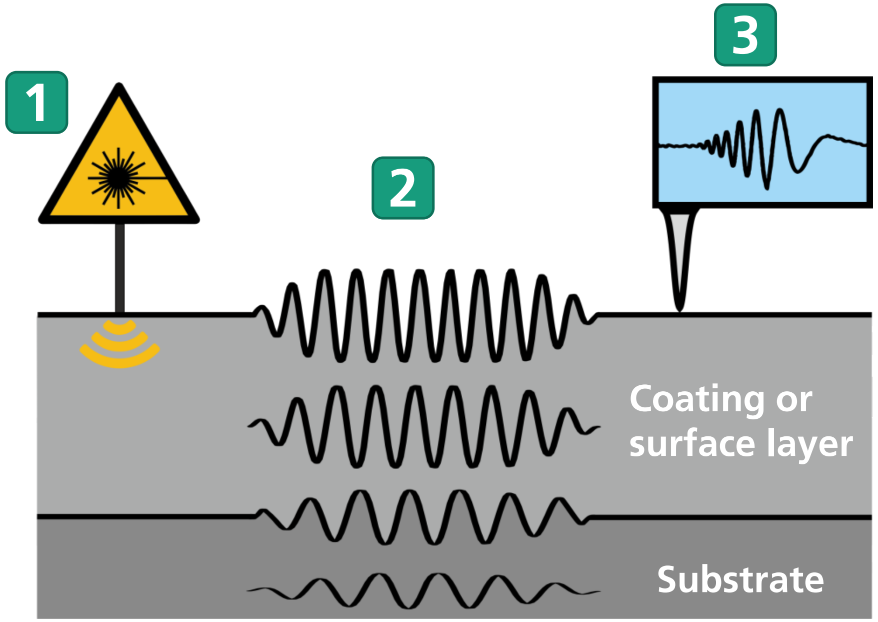
(5) Different analysis strategies

Possible results

- Young's Modulus
- Density
- Depth of: Nitriding layer, Case-hardening, Damage layers
- Porosity, crack density
- Delamination
- Build-up structure
- Surface hardness

…. Anything that affects your mechanical integrity of the material

Surface wave excitation and measurement



1 SAW excitation

- Broadband surface acoustic waves (SAW) induced by short laser pulses

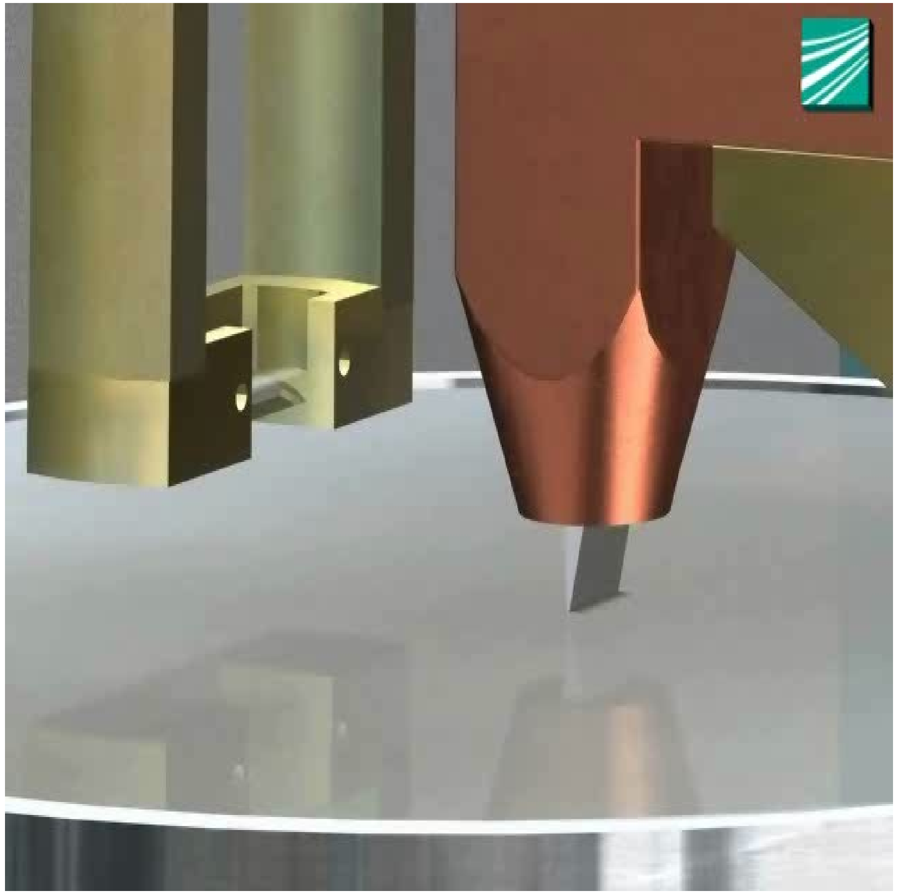
2 SAW propagation

- Penetration depth of SAW ≈ wavelength $\lambda = c/f$
- SAW velocity c depends on frequency f $c = c(f)$

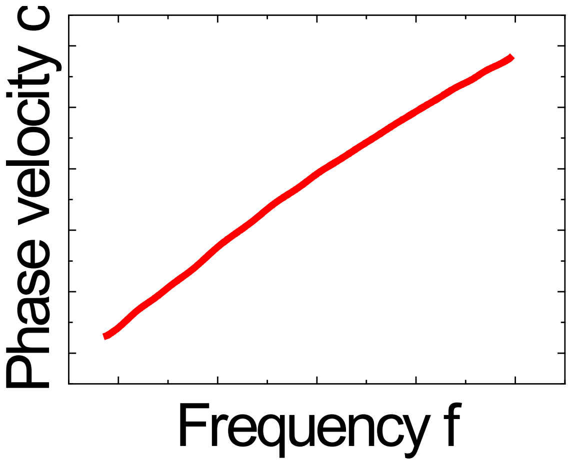
3 SAW detection

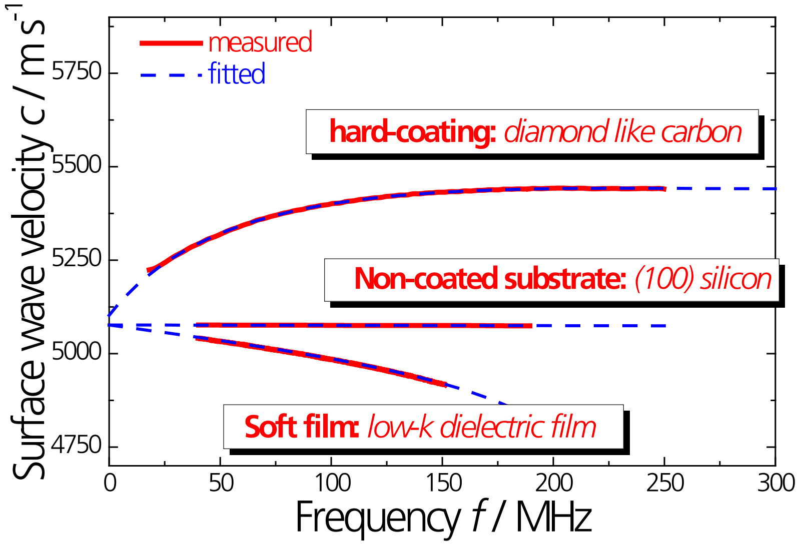
- Mechanical vibrations → electrical signals
  - Wedge type sensor with piezoelectric foil for 20-250 MHz
  - Conventional ultra sound sensor for 1-20 MHz
- Oscilloscope measures impulse run-time

Surface wave excitation and measurement - Video



Evaluation of Measurement

4 

5 

4 Measuring procedure and data analysis

- Variation of propagation distance *x*
- FT of the detected signals

→ Phase spectra *Φ(f)* for different distances and phase velocity *c(f)*

$$c(f)=\frac{(x_2-x_1)2\pi f}{\Phi_2(f)-\Phi_1(f)}$$

= dispersion curve

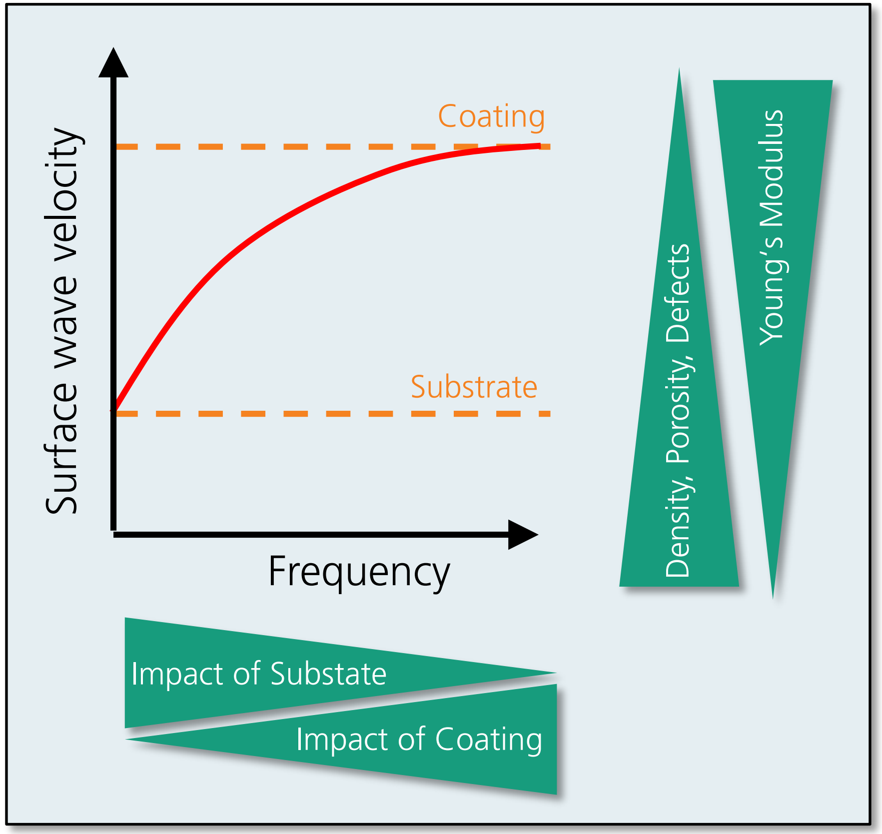
- Shape of the dispersion curve *c(f)* depends on elasticity, density and film thickness

5 Approaches to get film properties

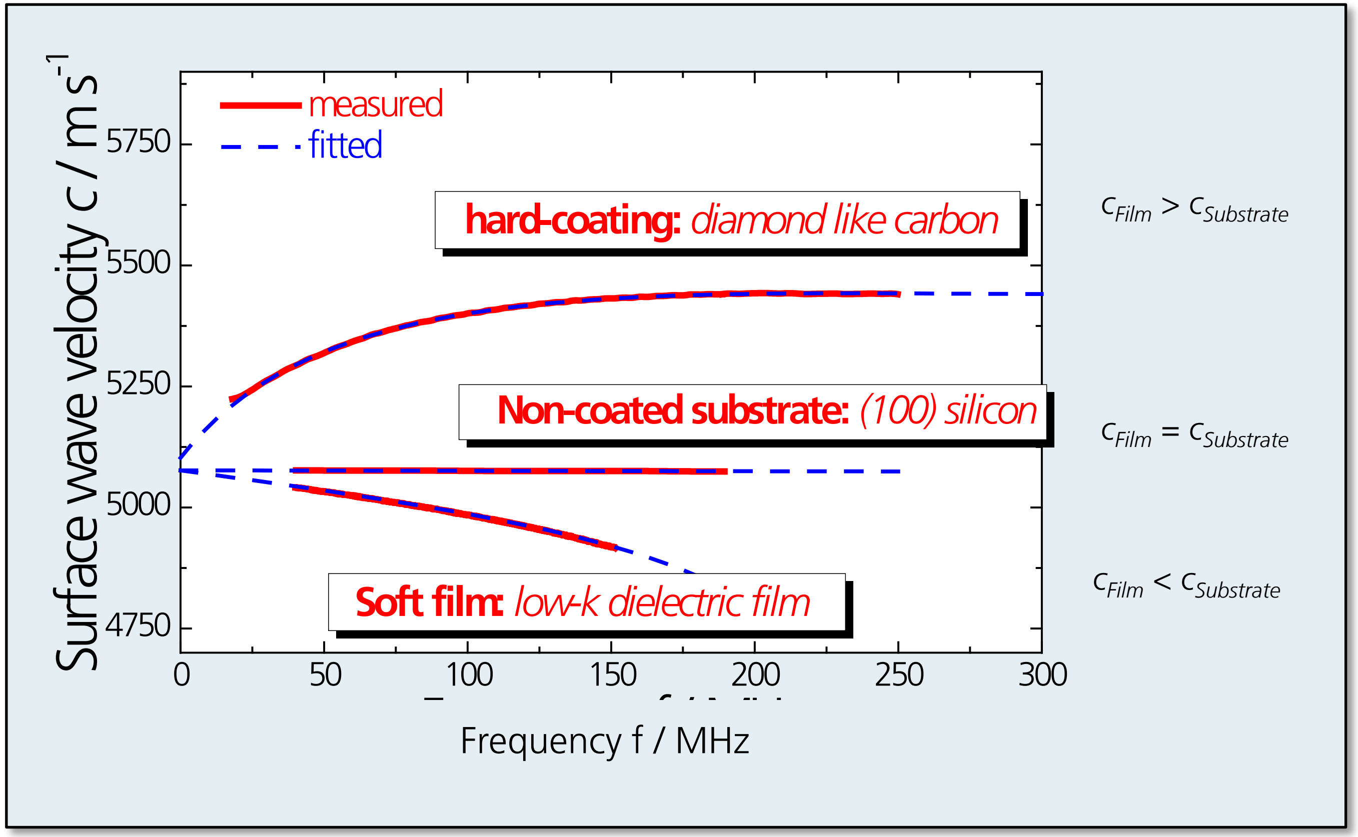
- Fitting measured curve to theory, using a material model
- Calibration with another method
- Defining ok/not ok boundaries from known samples
- Using regression fitting and KI with know samples

Dispersion Curve – Influence of Material System

Schematic influence



Actual influence measured on coated silicon wafer



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Dispersion Curve Analysis – Multilayer Material Model

Multilayer Model by Haskell and Thomson

- Is able to model SAW propagation for any multilayer stack consisting of homogeneous layers
- 1 to 3 material parameters can be obtained from fitting data to model
- Number of material parameters that can be fitted depend on curvature of dispersion curve
- Other parameters can be derived from data bases, independent measurement or assumption

film 5: E_5, v_5, ρ_5, d_5
film 4: E_4, v_4, ρ_4, d_4
film 3: E_3, v_3, ρ_3, d_3
film 2: E_2, v_2, ρ_2, d_2
film 1: E_1, v_1, ρ_1, d_1

substrate

isotropic: E, v, ρ
cubic: C_{11}, C_{12}, C_{44} , ρ

Theory of Thomson and Haskell

- Exact solution for dispersion curve of a stack of layers

Haskell; Bull. of the Seism. Soc. of America 43, 1953.
Thomson; Journal of Applied Physics 21, 1950

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Dispersion Curve Analysis – Number of independent parameters

Material

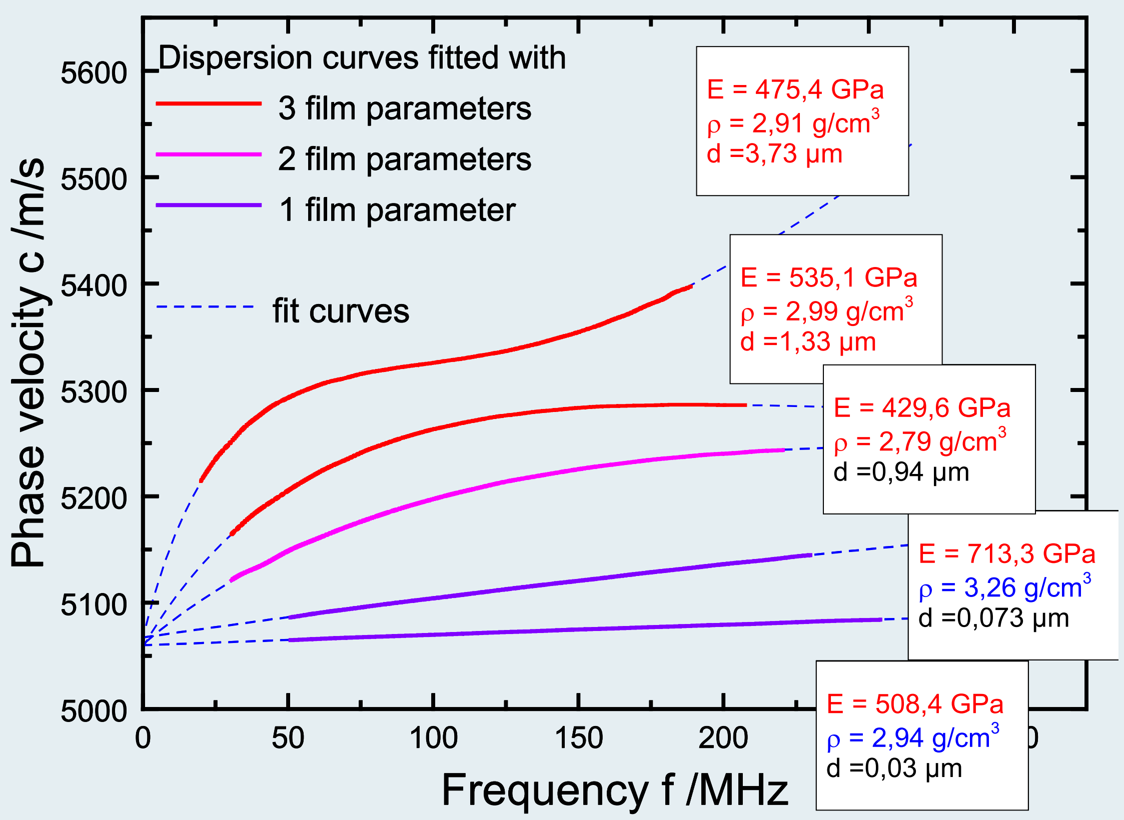
- Coating: ta-C = superhard amorphous carbon
- Substrate: Si wafer

Film parameters that can be measured

- Young's modulus E
- Density ρ
- Film thickness d

More coating parameters can be fitted for

- High differences of coating and substrate
- High frequency range



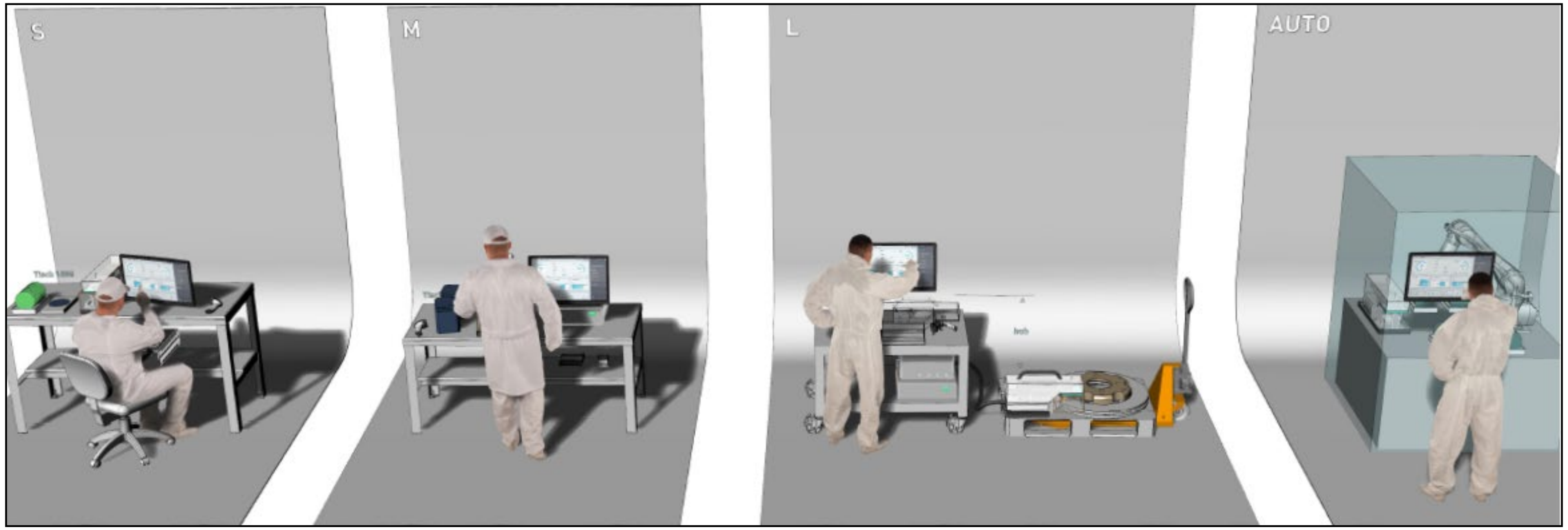
M. Leonhardt, Fraunhofer IWS Dresden

Development and Background

Current Developments

Development topics

- Quality control suitability: automated measurement and evaluation functionality
- Mobile head for robot or hand for measurement on large parts
- Measurement at elevated temperature
- Integration for customer-specific applications



Scaling concept for LAwave system technology

From left to right

- Fully manual operated R&D tool
- Half-automated quality control system
- Quality control system for large components
- Fully automated quality control tool

History of System Development

Case Studies

Application - Overview

Young's modulus, thickness, density of

- All kinds of coatings: PVD, CVD, spin coating, thermal-spraying, cladding, electroplating, …
- E.g. amorphous carbon coatings (DLC), nitrides, carbides, oxides, other ceramics
- Metal films
- Low-k films
- Polymeric sensor films
- Bulk materials, e.g. steel, brass, cemented carbide
- Si, GaAs semiconductors

Depth of

- Subsurface damage from silicon wafer processing
- Surface hardening zones e.g. after metal finishing

Case study: Very thin films < 10 nm

Material

- PVD coatings with thickness < 10 nm

Results

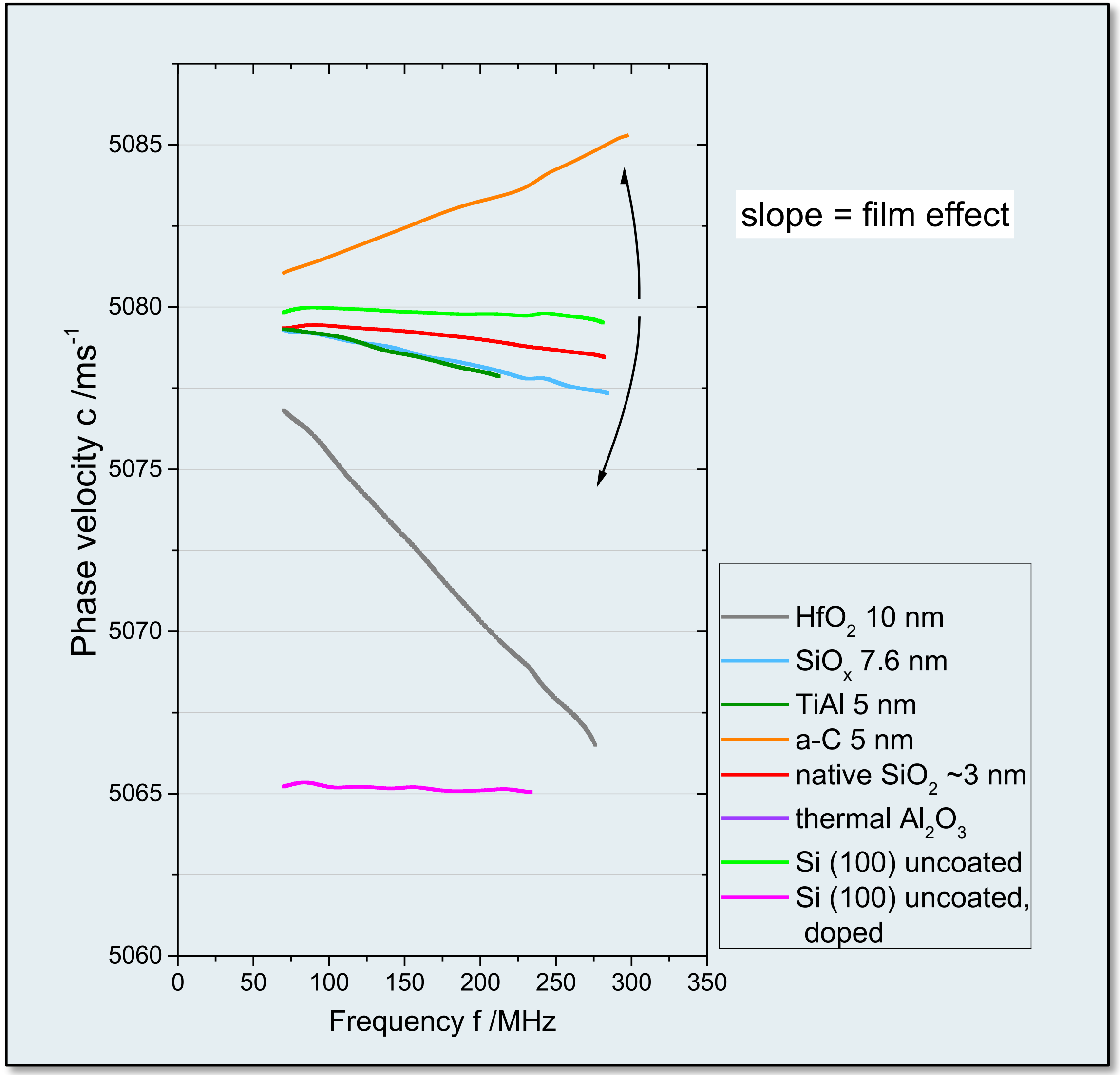
- Measurement of Young's Modulus

HfO_2	220.4 GPa
Native SiO_2	39.8 GPa
SiO_x	41.7 GPa
a-C	373.4 GPa
TiAlN	142.8 GPa
Silicon wafer	165.2 GPa (C11)
Silicon wafer (high doping)	162.9 GPa (C11)

- Measurement of thickness of $Si/Al/Al_2O_3$ multilayer stack

Thermal Al2O3	3.9 nm

➔ **Possibilities beyond nanoindentation**



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Case study: Quality control of superhard carbon coatings

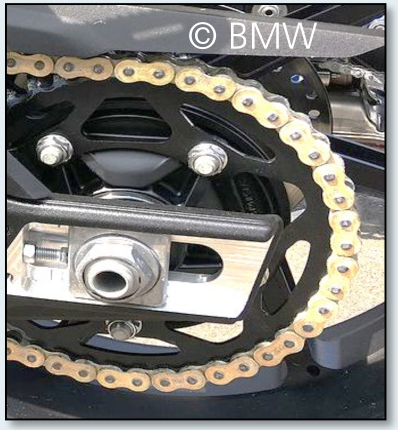
Material

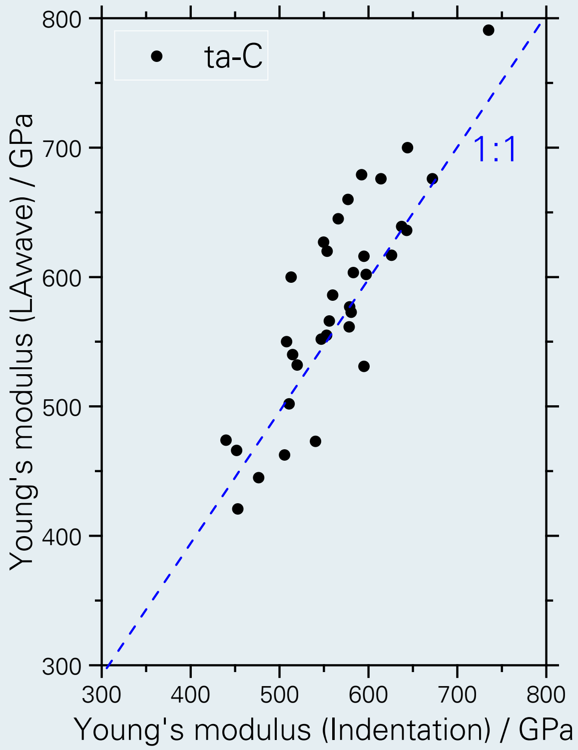
- Superhard amorphous carbon coatings (ta-C, H-free DLC), hardness 40..70 GPa
- Application: Low-wear low-friction coating, e.g. piston pins in ICE, motorcycle chain
- State-of-the art: Nanoindentation → slow and error-prone technique with high indenter wear

Results

- LAwave allows to access
  - Coating modulus, coating hardness
  - Coating thickness

in less than 60 seconds





Case study: Lateral cracks in SHVOF coatings

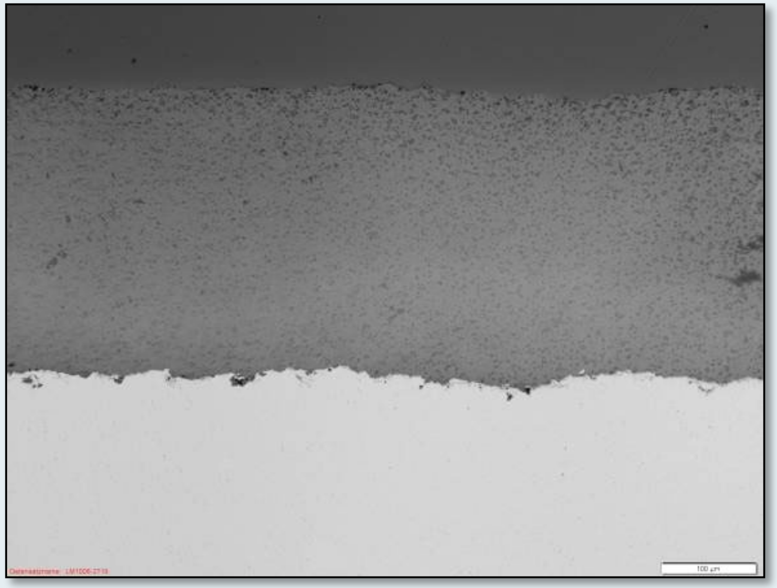
Material

- Al_2O_3 SHVOF sprayed
- Thickness around 400 µm
- Coating structure: homogenous, risk of lateral cracks

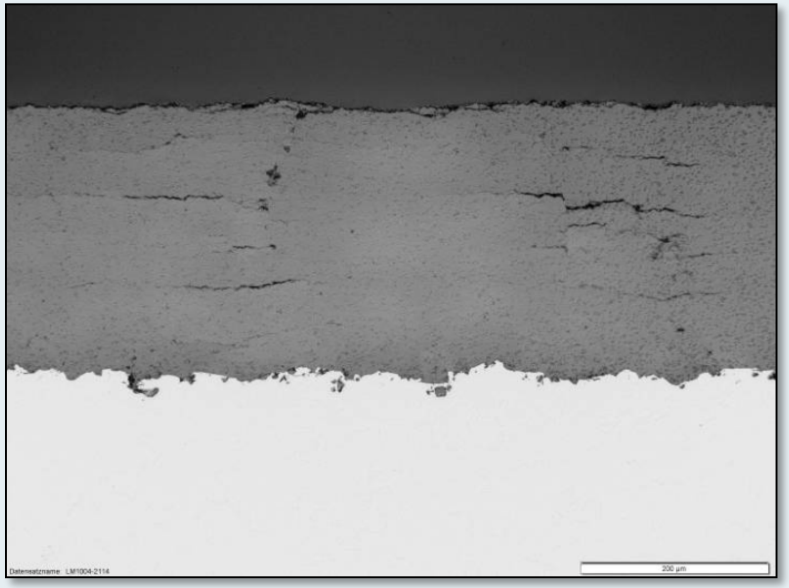
Results

- Measurement of elastic modulus
- Elastic modulus decreases due to lateral cracks

➔ **Non-destructive measurement of critical defects**



Dense structure
E = 101 GPa



Lateral cracks
E = 46 GPa

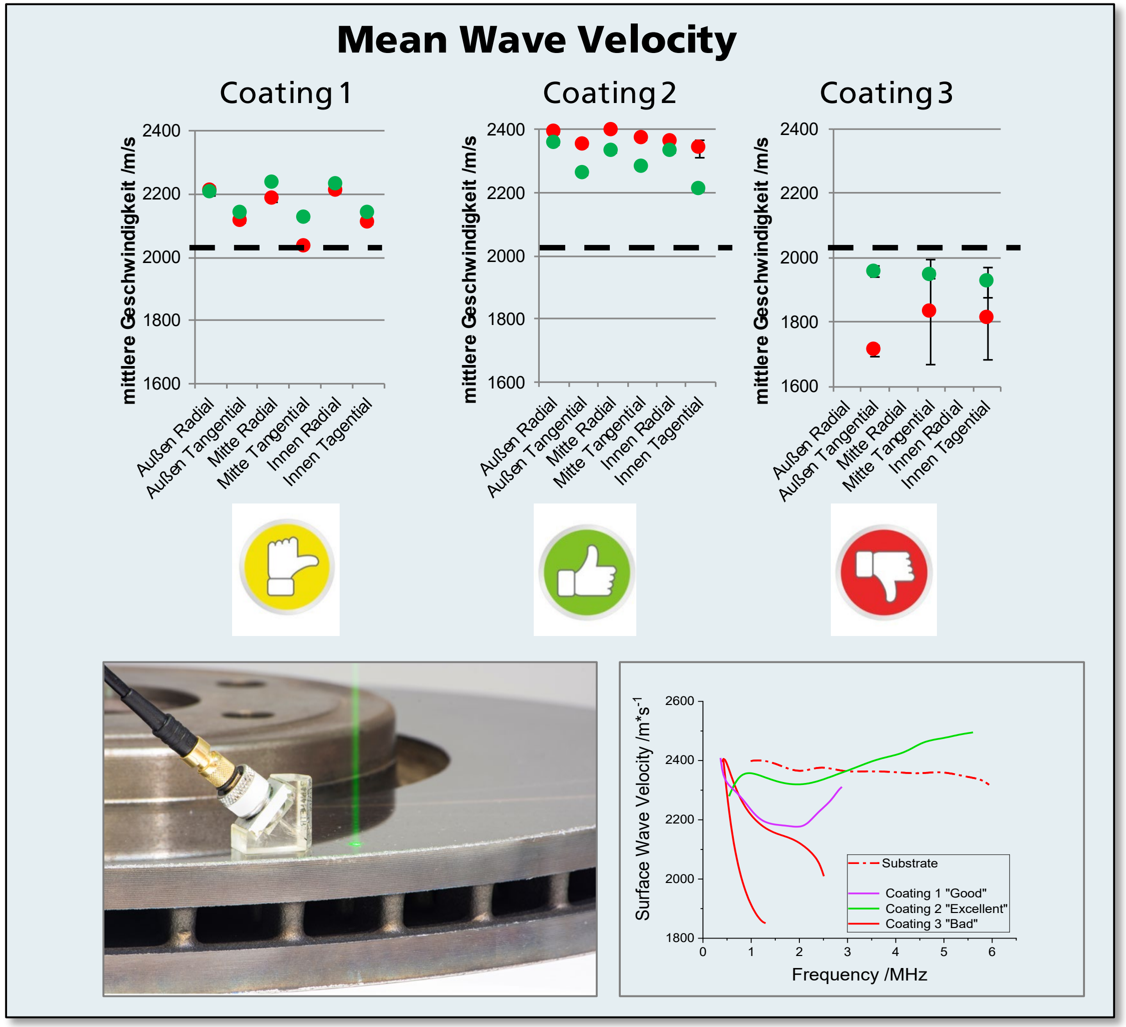
Case study: Development of novel brake disk coatings

Material

- Multilayer coatings from high speed laser cladding, carbides in Fe-based matrix
- Application: Novel brake disk coatings for high performance and e-mobility
- State-of-the-art: Cross section + SEM imaging → time consuming (~ hours… days), expensive, big infrastructure

Results

- LAwave measures mechanical key features
- Front and back, ⊥ and || to deposition direction, anywhere on the disk
- Non-destructive (disk can be measured before and after test bench)
- six representative spots measured in less than 30 minutes



Case study: Defects in APS-Al_2O_3

Material

- Spray technologies: APS (or HVOF, …)
- Al_2O3 (or Cr2O3, TiO2, …)
- Thickness 100 to 600 µm
- High roughness Ra > 1 µm
- Coating structure: micro-cracks and porosity

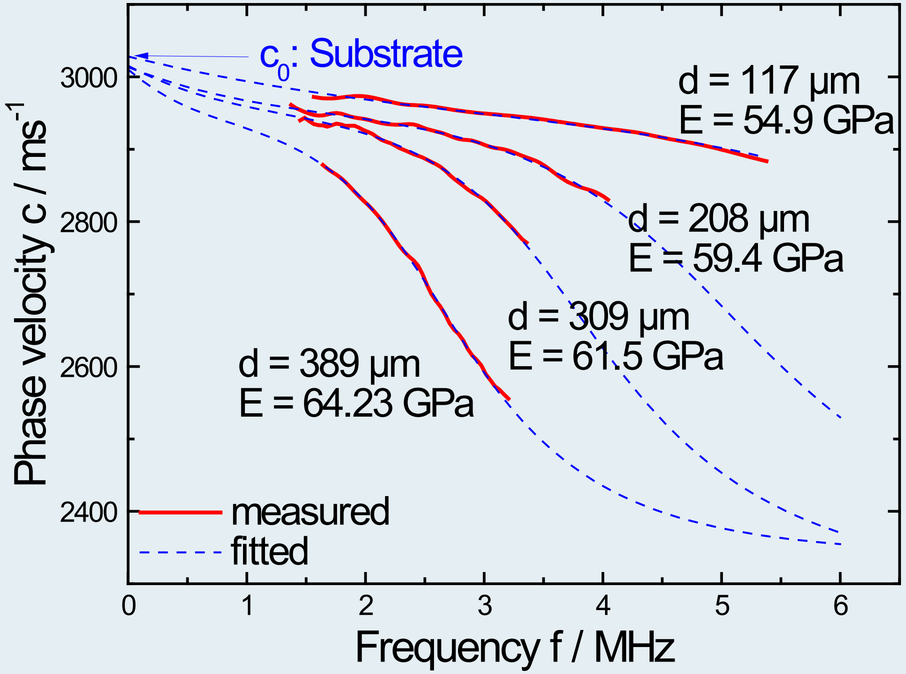
Results

- LAwave measurement gives coating thickness and effective elastic modulus E
- Effective elastic modulus varies due to different crack and pore density

➔ **Quality and mechanical behavior of coating can be measured non-destructively**

Al_2O_3 (APS) on steel

E (tabulated; compact material) = 350 GPa



L.-M. Berger et. al.: VIP-Journal Vol. 24, 2012

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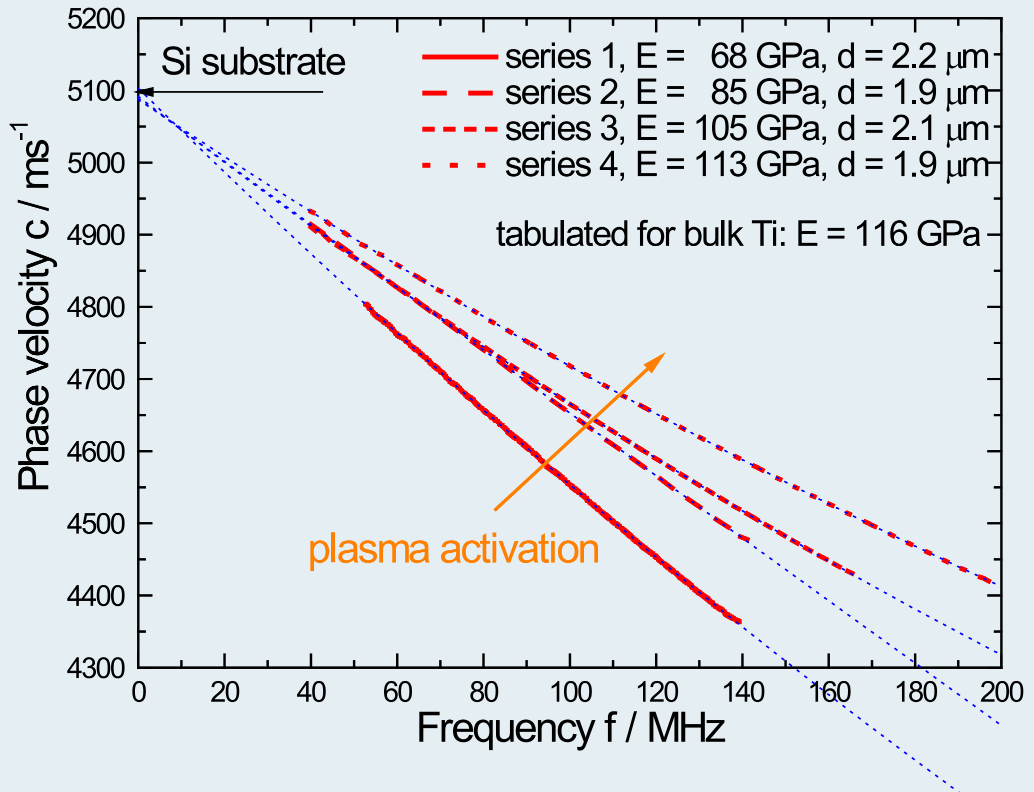
Case study: Pores in metal films (1/2)

Material

- 2 µm Titanium coating on Si wafer
- PVD: Electron beam evaporator + additional plasma activation

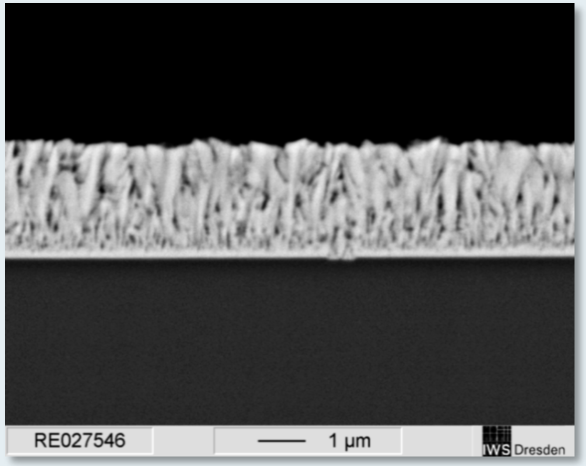


M. Leonhardt et. al.: Surface and Coatings Technology 2004

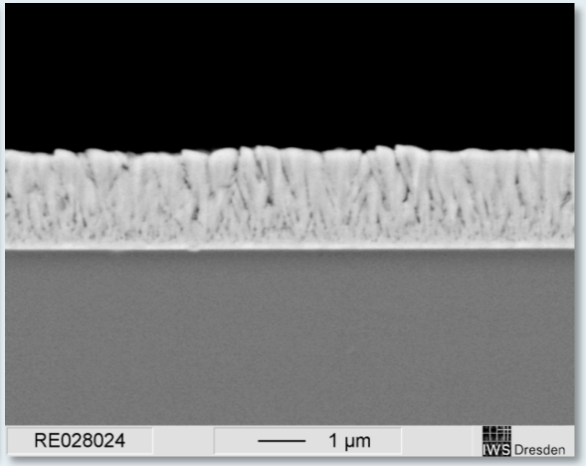
Results

- Effective Young's Modulus is measure of porostiy
- No activation → porous films (E = 68 GPa)
- High activation → dense films (E = 113 GPa)

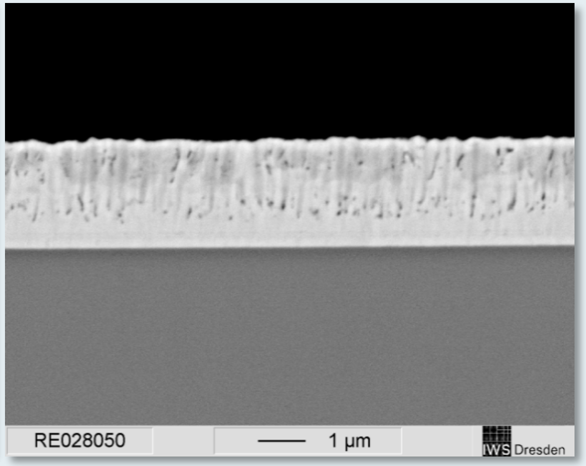
Example: Pores in metal films (2/2)



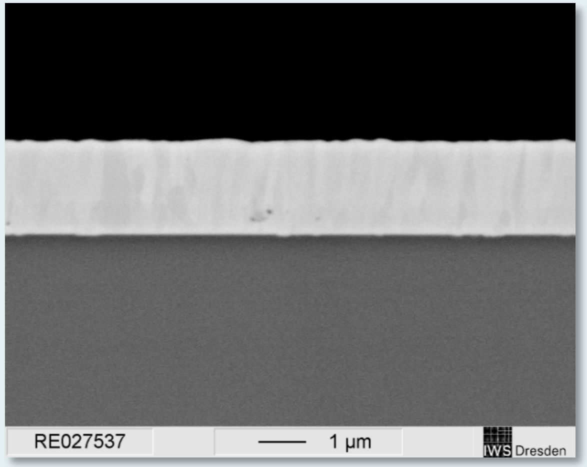
Series 1
E = 68 GPa



Series 2
E = 85 GPa



Series 3
E = 105 GPa



Series 4
E = 113 GPa
(Compact Ti: E = 116 GPa)

M. Leonhardt et. al., Surface and Coatings Technology 185 (2004) 292

➔ **Effective Young's Modulus strongly correlates with porosity observed in SEM cross section**

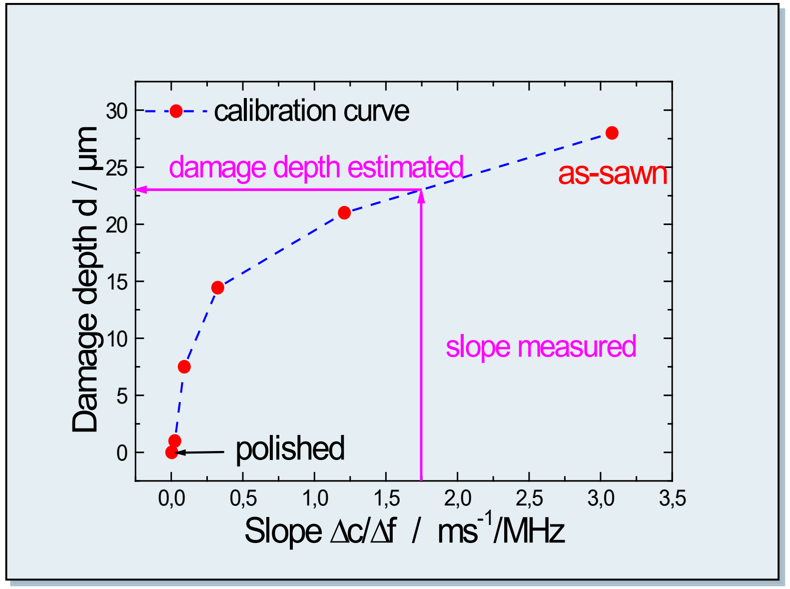
Case study: Subsurface damage in semiconductor wafers

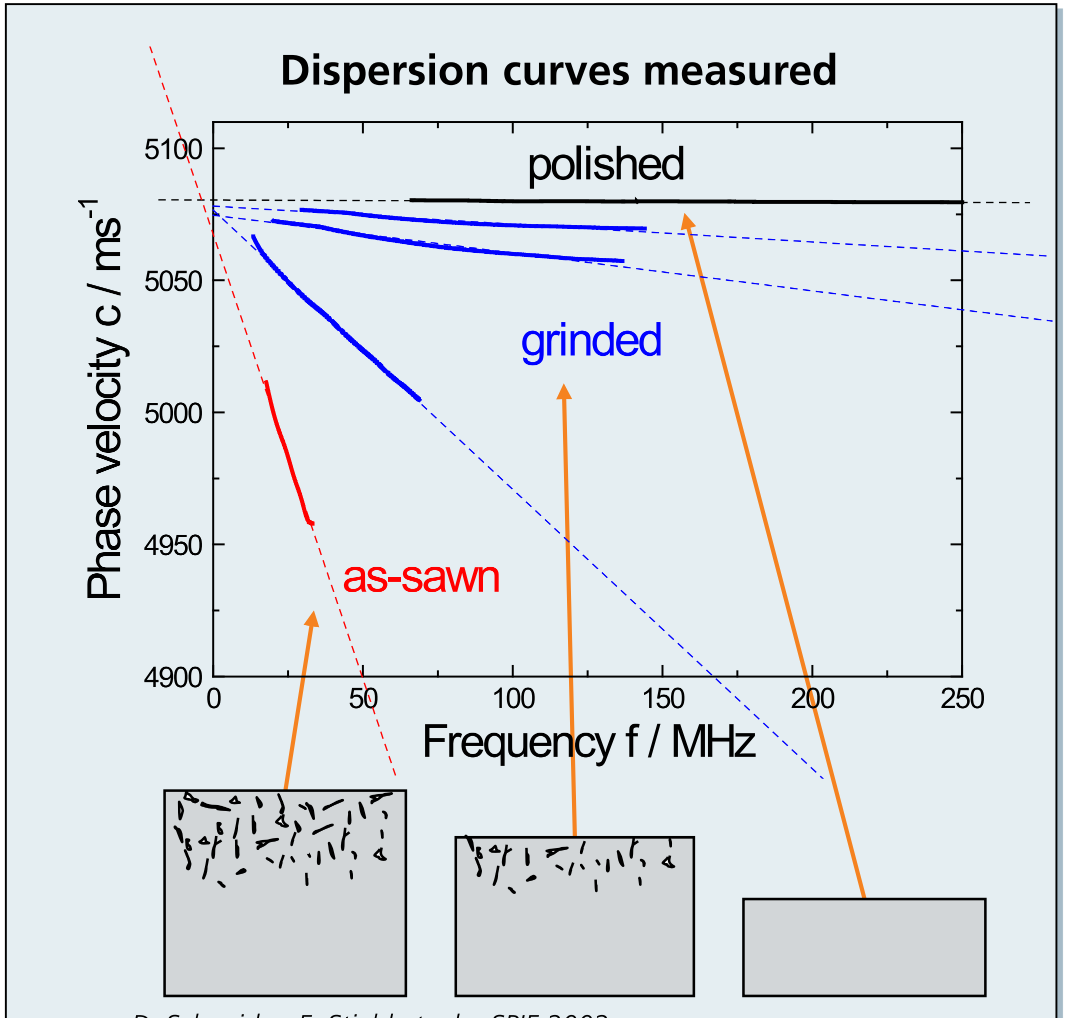
Material

- Semi-conductor surfaces, damaged from processing

Results

- Damage layer ➔ dispersion
- Slope = damage layer depth ➔ allows quantification





D. Schneider, E. Stiehl et. al., SPIE 2002

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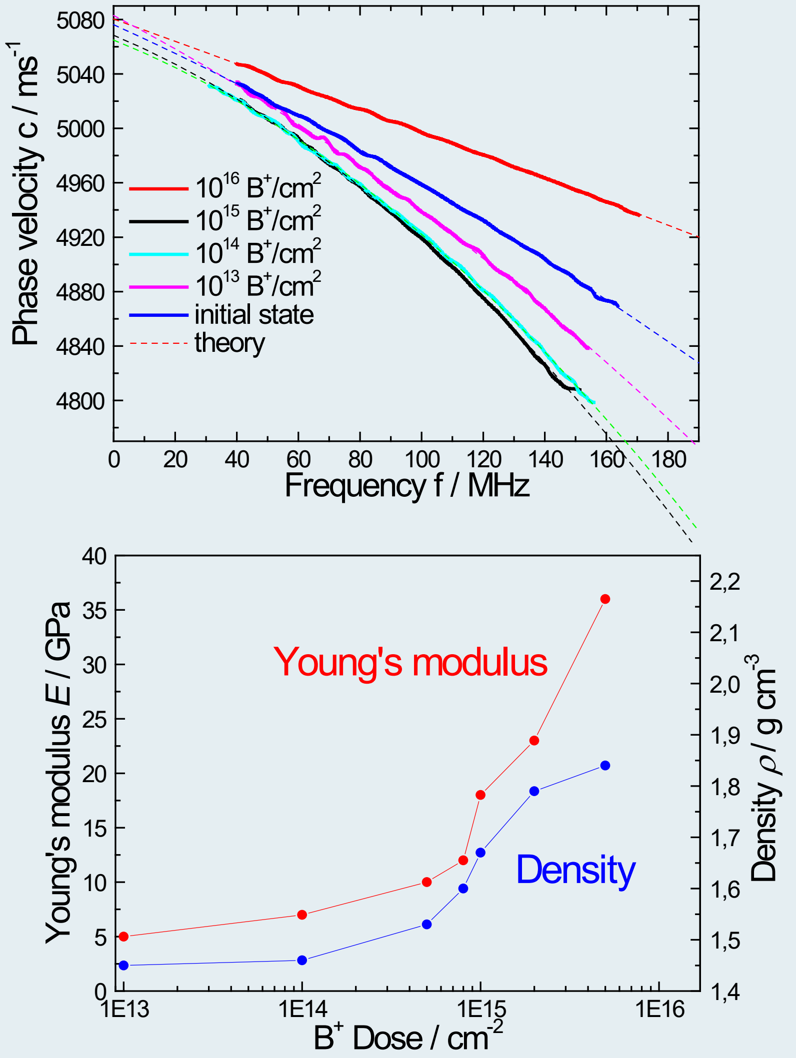
Case study: Polymeric sensor films

Material

- Polyimide films on silicon wafer for humidity sensors
- Film thickness 500 to 600 nm
- B^+ ion implantation to improve sensor properties

Results

- Young's modulus E and Density ρ were obtained from the measurement
- Density and Young's modulus increase with B^+ dose
- Distinct effect for a B^+ dose > 1015 B^+/cm^2
- Young's modulus increased by approx. 700 %



M. Guenther et al. Macromolecular Symposia 212 (2004) 245

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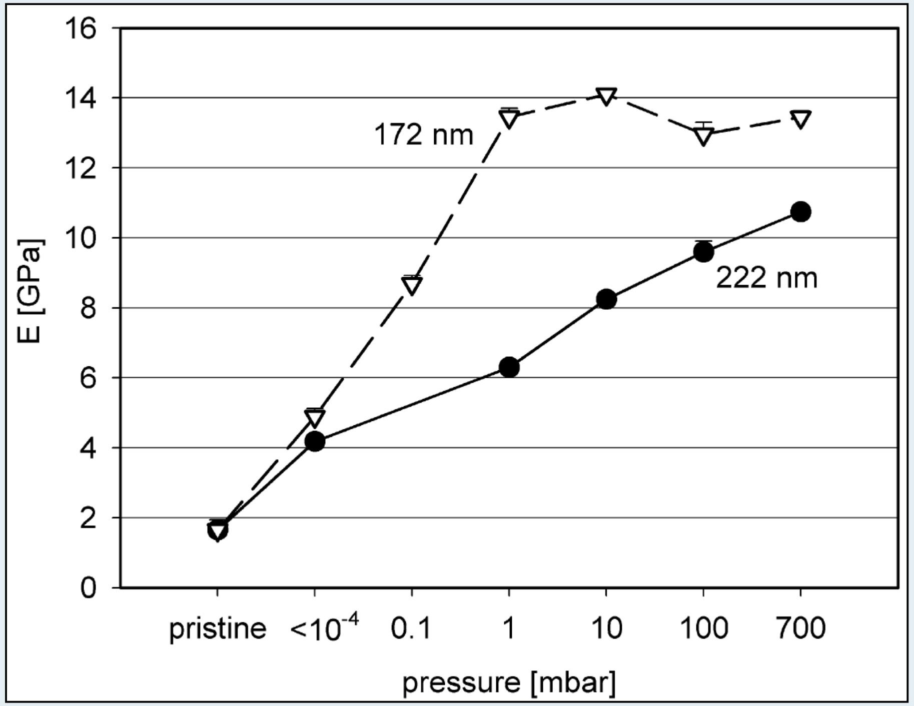
Example: Porous low-k films

Material

- Nano-porous SiCOH low-k films
- High porosity: > 40 %
- Rel. permittivity k < 2.5
- Minimum required stiffness E > 5 GPa

Results

- Young's modulus and density can be measured
- Higher reliability than results from nanoindentation



Irradiation with 172 nm and 222 nm photons, ρ = 1.2 gcm^{-3} and d = 200 nm

Prager et al. Microelectronic Engineering 85 (2008) 2094–2097

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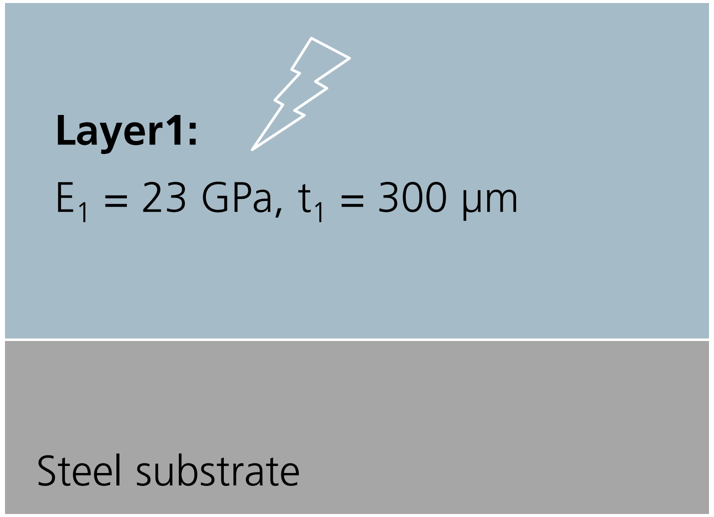
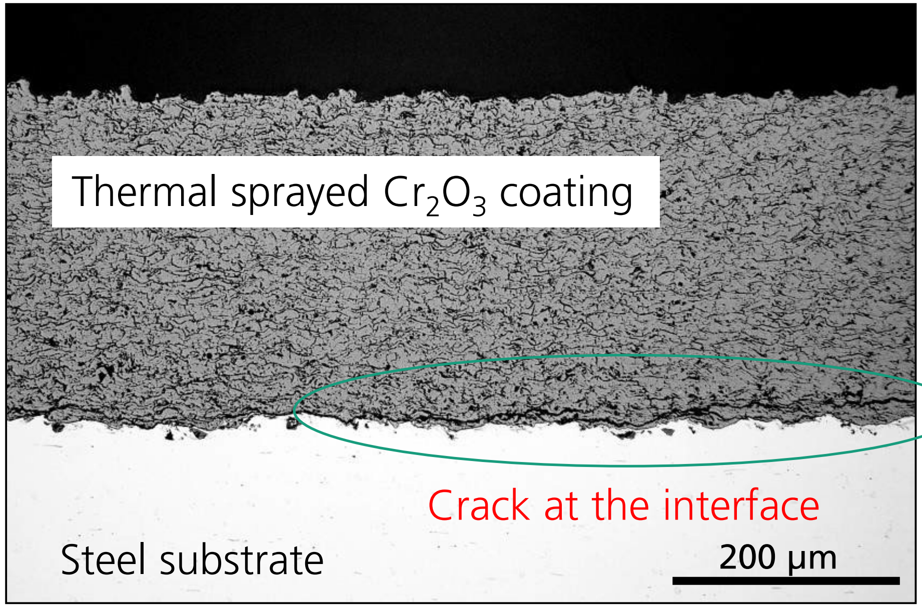
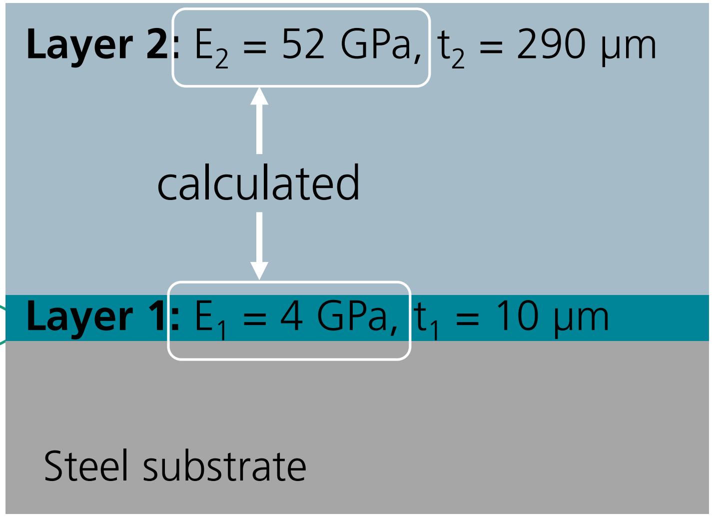
Case study: Delaminations







1st step: simple 1-layer model

- Measured Young's modulus smaller than expected (E = 50 GPa)

➔ Measurement and model do not fit

2nd step: cross section preparation

- Delamination revealed at interface

3rd step: 2-layer model

- 2-layer model fits expectation when weak interface is assumed

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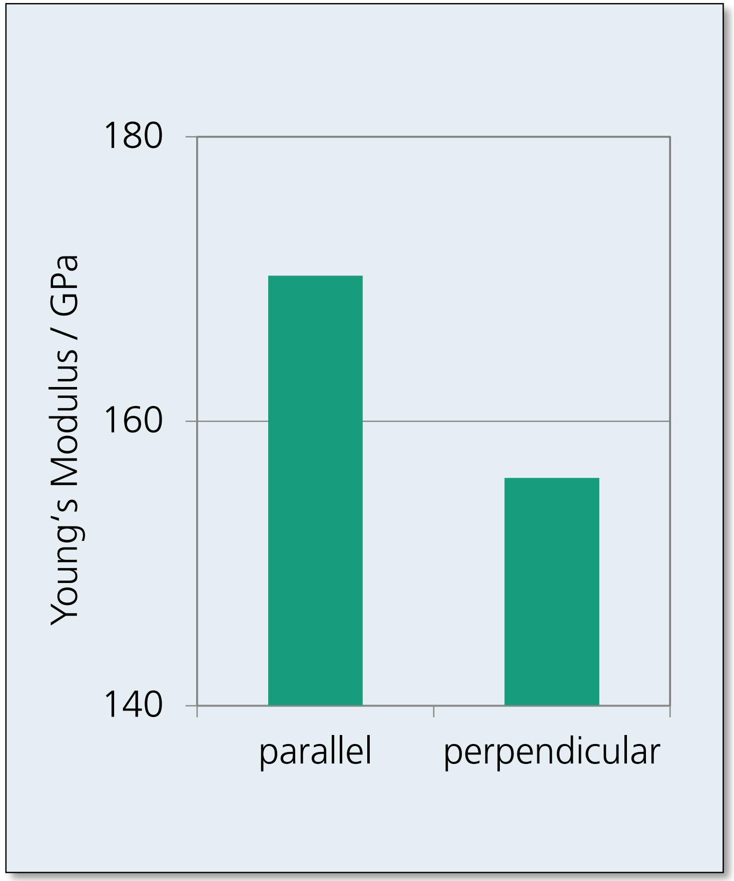
Case study: Laser cladding, laser buildup welding

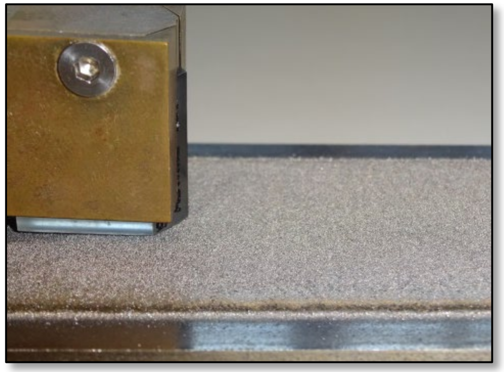
Material

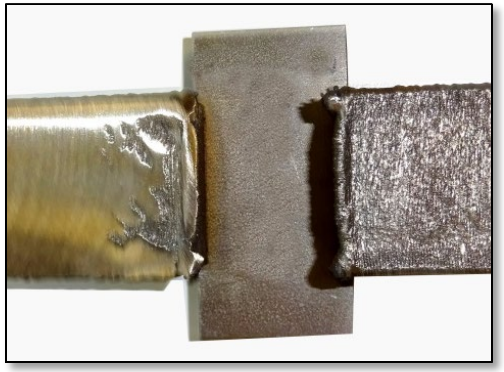
- Coatings from Laser Cladding on steel, thickness: 0,5 … 2 mm
- Bulk samples from Laser Buildup Welding
- e.g. Inconel 625, 316 L
- High roughness Ra > 1 µm

Results

- Young's Modulus from measurement
- Influence of buildup direction (⊥ or || to cladding lines)
- Microstructure: Influence of cracks and porosity







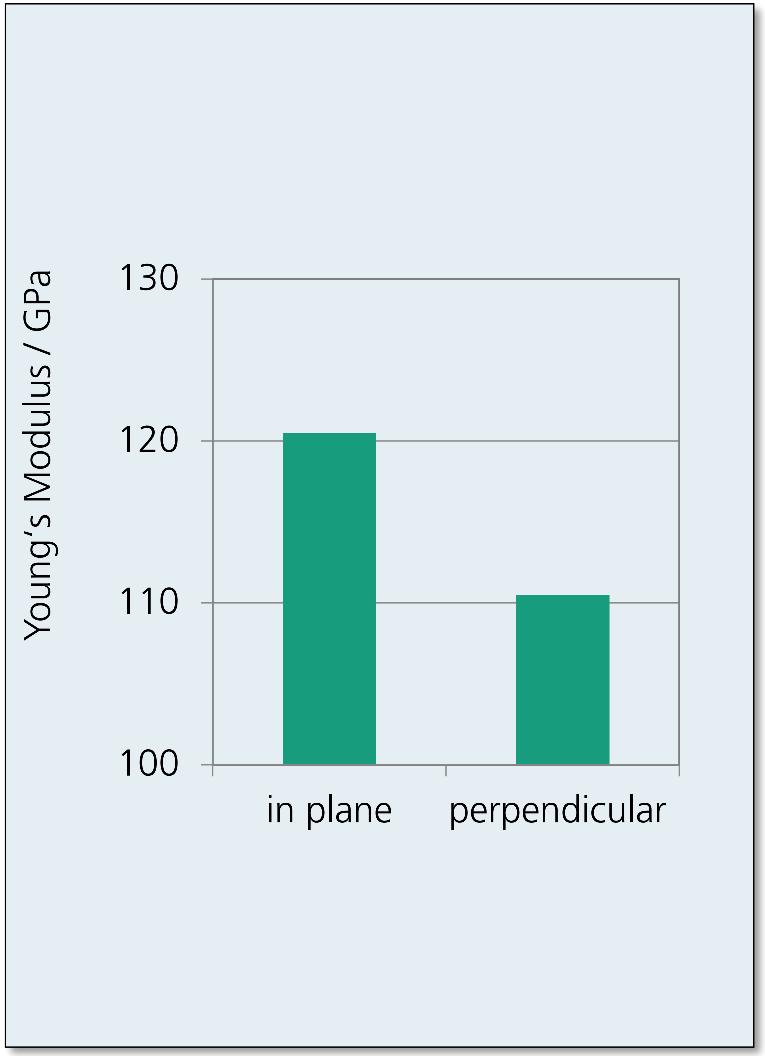
Case study: Parts generated from Selective Laser Melting (SLM)

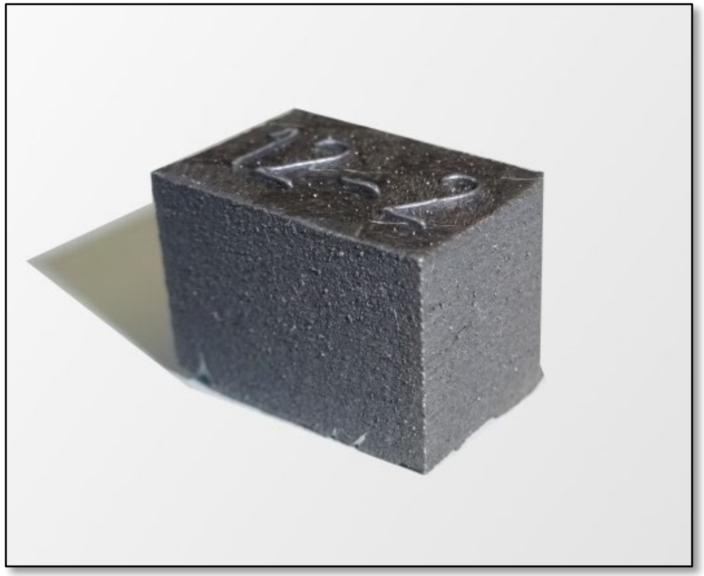
Material

- Parts generated by selective laser melting
- Material: e.g. AlSi40, Ti6Al4V, …

Results

- Young's modulus
- Influence of buildup direction (⊥ or || to built up lines)
- Microstructure: Influence of cracks and porosity

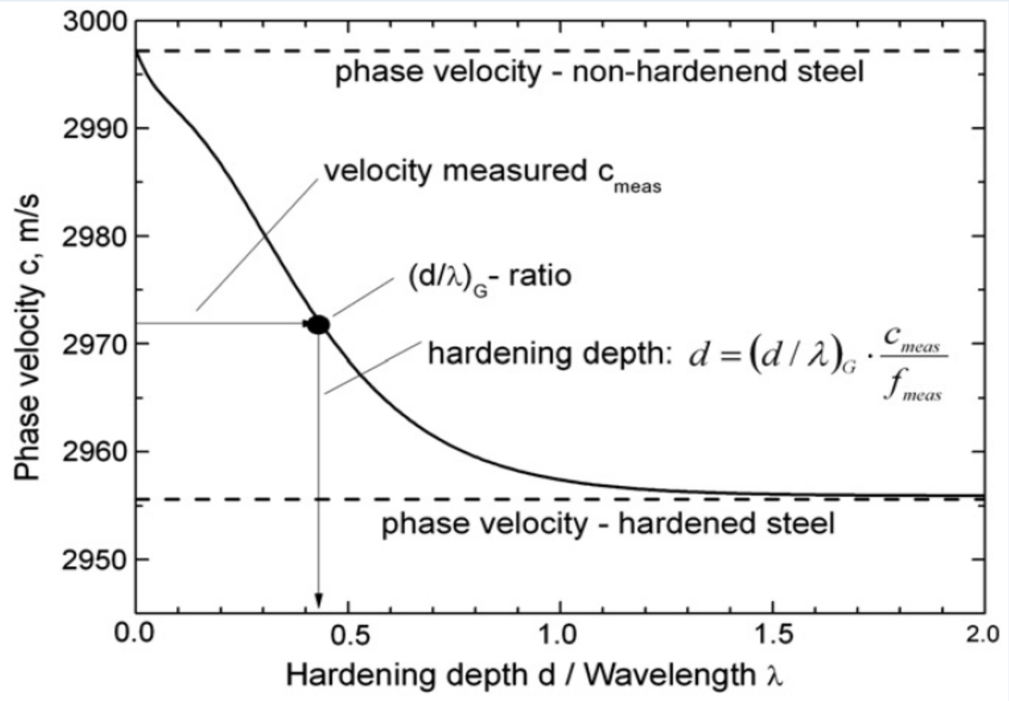




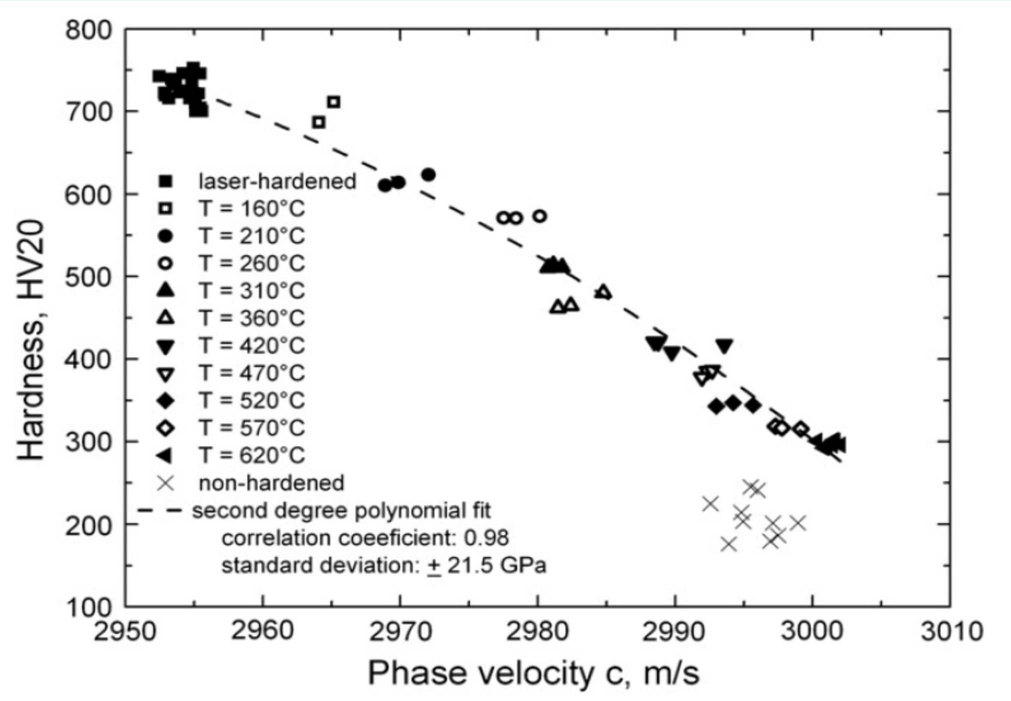
Case study: Hardening depth

Material: Surface hardened metal (case hardening, laser hardening, nitrogen hardening, …)

Results: Hardening depth, surface hardness



D. Schneider et al, Surface and Coatings Tech., 206, p. 2079-2088, 2012



D. Schneider et al, Surface and Coatings Tech., 206, p. 2079-2088, 2012

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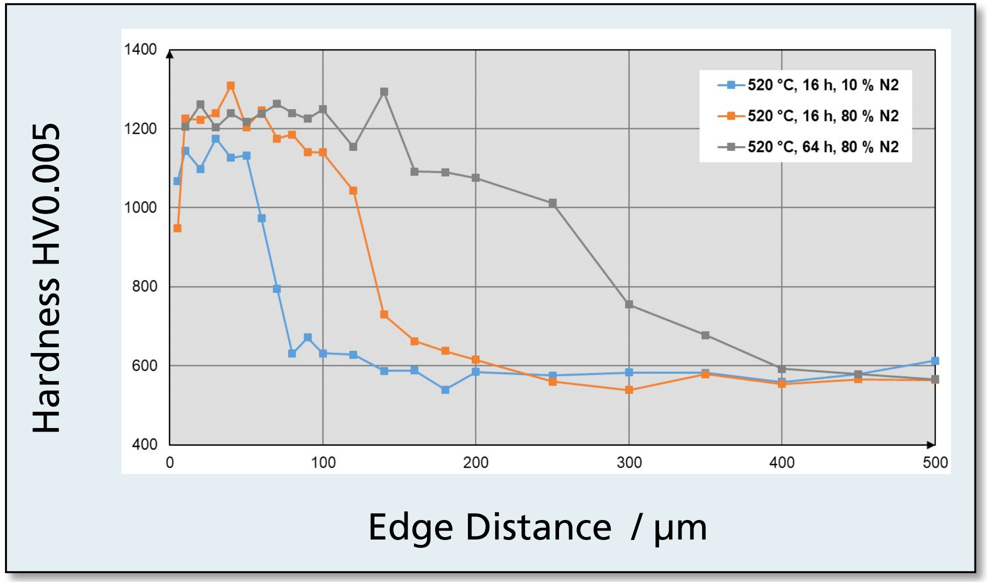
Case study: Nitriding depth

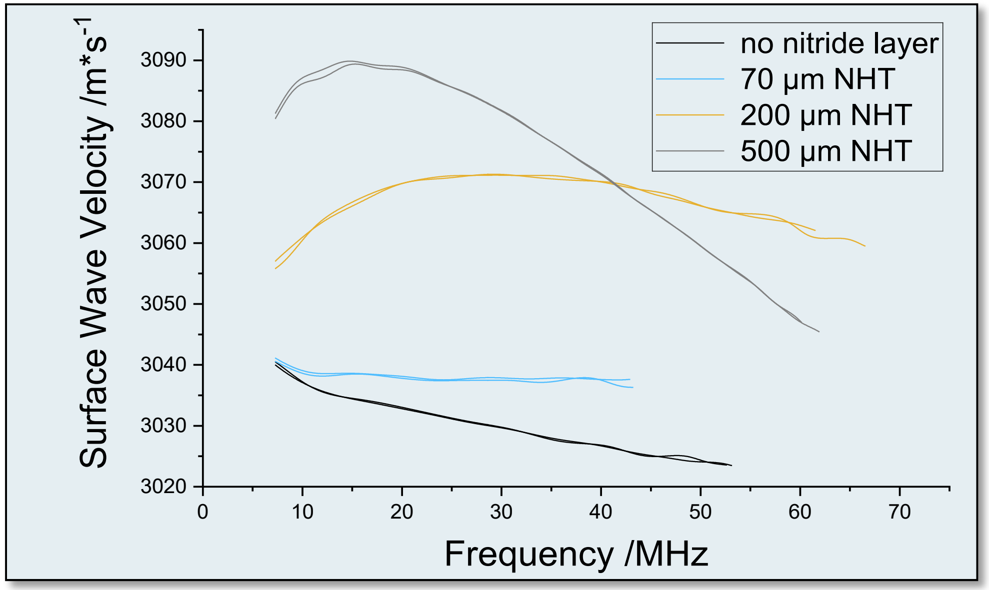
Material

- Steel 1.2343
- Nitrided with different nitride hardening depths (= NHT)

Results

- Strong correlation between hardness profile and dispersion curves
- Dispersion curves hold information about NHT, surface and core hardness, and more





Methodical Aspects

Measurement on native rough surfaces

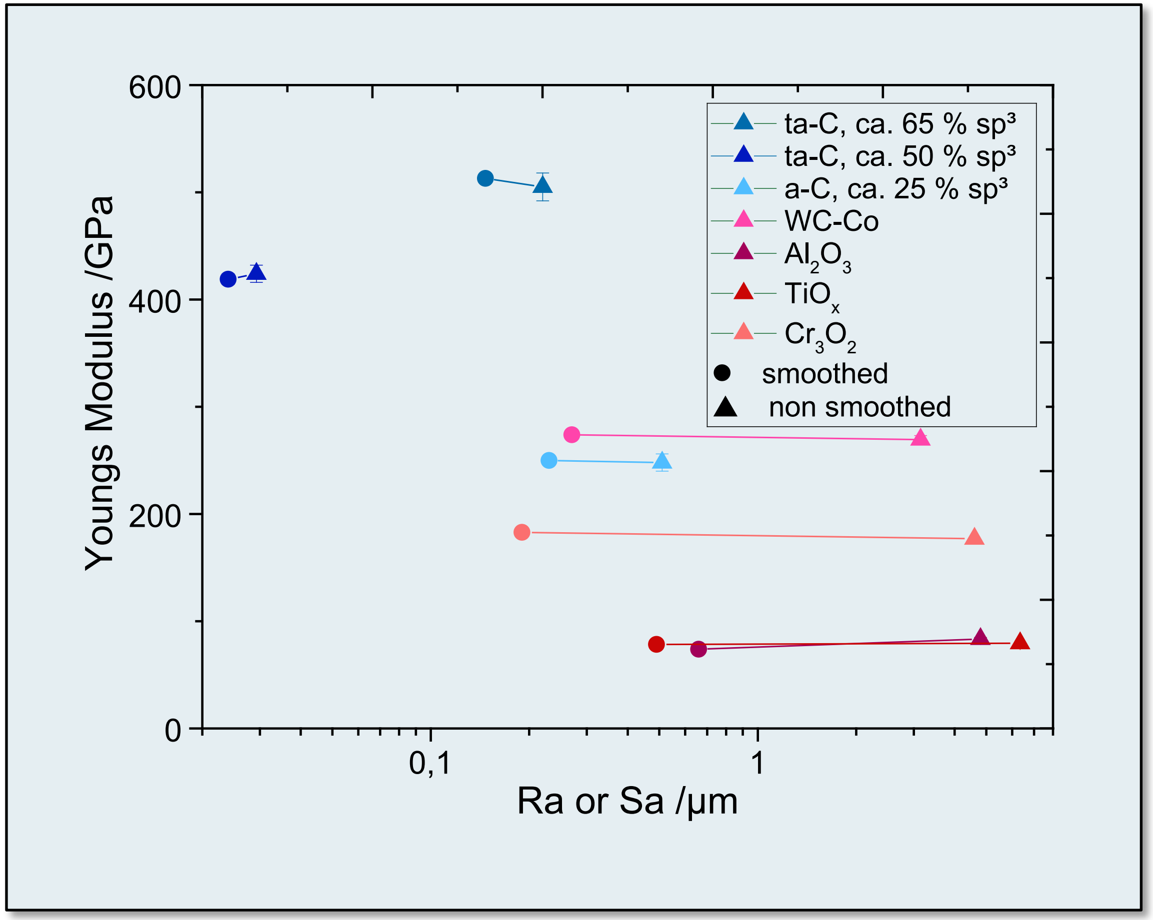
Material

- Various hard PVD and thermal spray coatings
- Surfaces both as-deposited and smoothed

Results

- Measurement on both surfaces conditions possible
- Young's Modulus does not change
- Condition: Roughness (Ra 0,02 - 6,5 µm) << wave length (ca. 50 µm @ 60 MHz)

➔ **Measurement on native rough surfaces as reliable as on smooth surfaces**



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LAwave Fraunhofer IWS

Influence of sample curvature

Measurement in axial direction

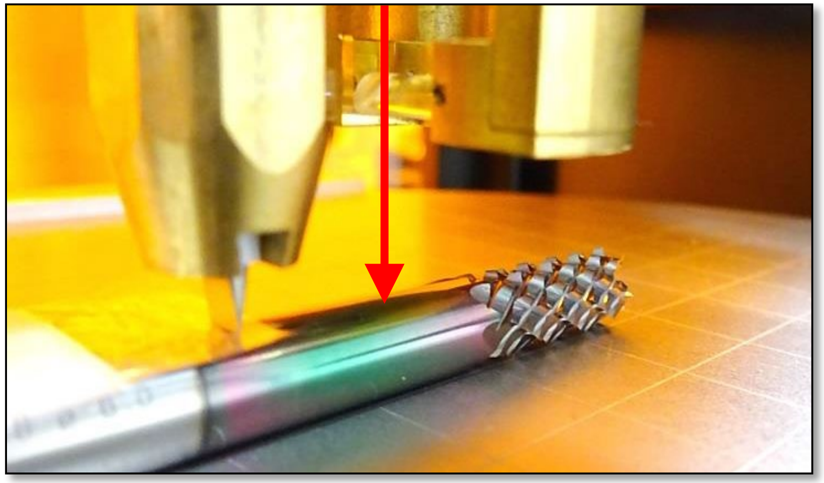
- No limitations from curvature
- Signal/noise ratio smaller

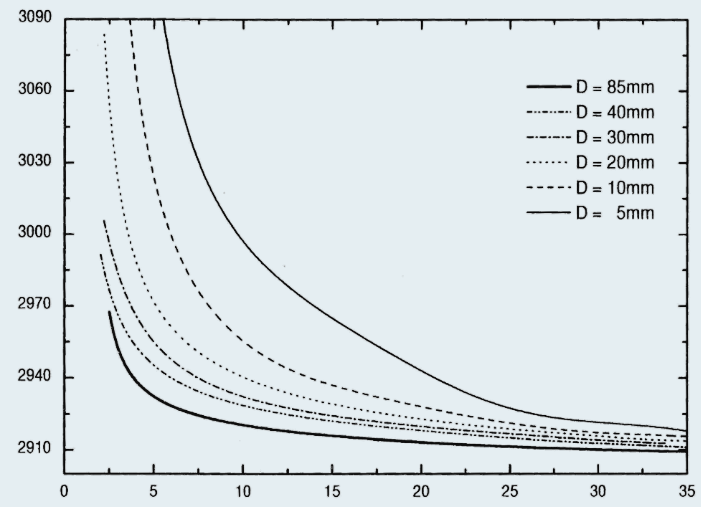
Measurement in radial direction

- Additional dispersion from curvature at low frequencies
- Correction of the influence of curvature mathematically possible

➔ **No general limitations from sample curvature**

➔ **Practical limitations for complex 3D structures**





J. Hennig, Thesis, WH Zwickau, 1998

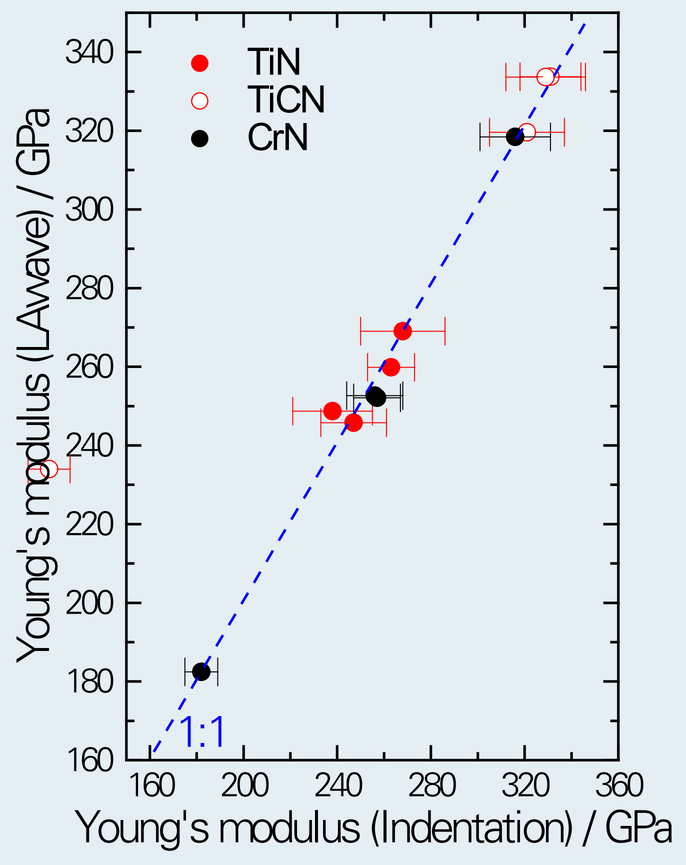
Comparison with instrumented indentation testing

Coating Materials

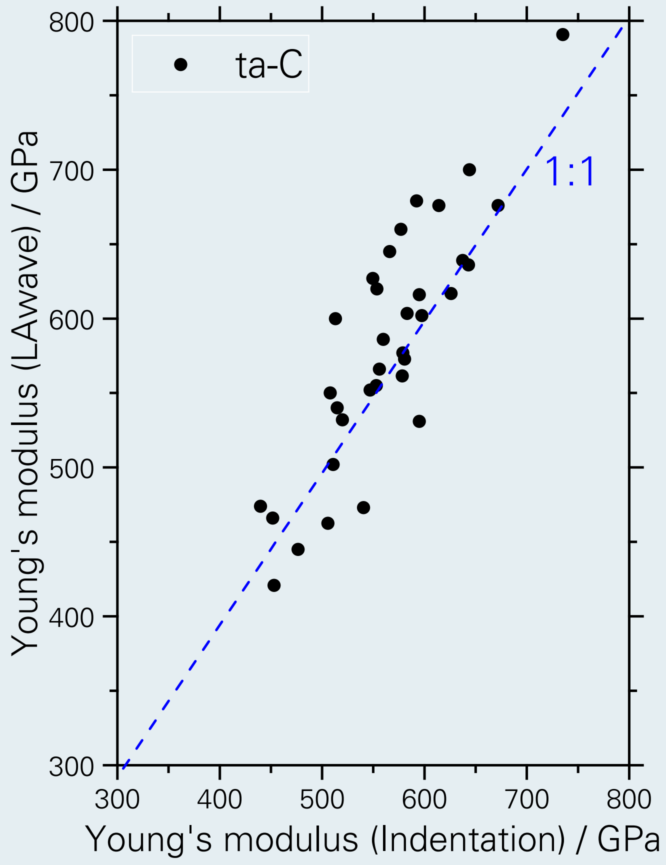
- TiN, TiCN, CrN (magnetron sputtering)
- ta-C (LaserArc)
- Film thickness: d >1 µm

Result

- Excellent agreement of Young's Modulus from both methods for solid, non-porous bulk materials



Schneider, Schultrich et al.: Thin Solid Films 1998



Zawischa, Lorenz; Fraunhofer IWS 2017

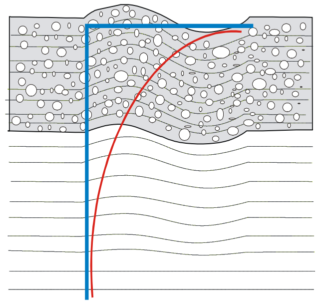
Comparison with instrumented indentation testing

Coating Materials

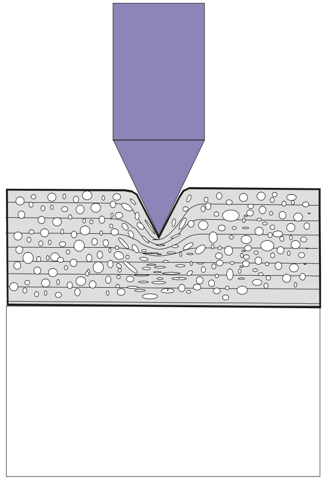
- Porous low-k films

Result

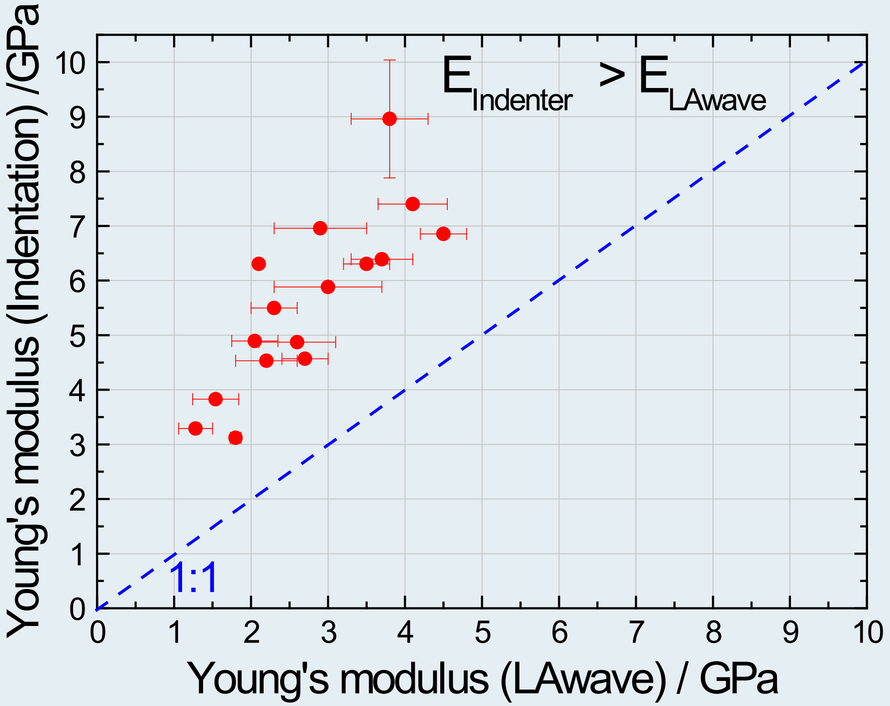
- Effective modulus is strongly overestimated with indentation due to compressed pores



Surface acoustic waves
Reversible deformation
➔ True elasticity



Indentation
Densification of microdefects
➔ Distorted results



S. Frühauf, University of Chemnitz

This example: $E_{Indenter} \approx 2 \cdot E_{LAwave}$

Comparison with instrumented indentation testing (nanoindentation)

	LAwave	Nanoindentation
Method	Dynamic: Sound velocity $c \sim \sqrt{(E/\rho)}$	Quasi-static: $E_r \sim dP/dh$
Measuring area	> 5 x 5 mm² (integral method)	< 10 µm² (local method)
Measuring time	One minute	~ 1 hour (including sample preparation and calibration)
Minimal film thickness	A few nanometers	≈ 100 nanometers
Surface roughness	No requirements	Smooth surface necessary
Difficult material systems	Transparent and high damping materials	Soft and superhard materials, very thin coatings

➔ **LAwave method has superior benefits over nanoindentation for many application scenarios**

LAwave around the world

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